**Organics by Gas Chromatographic/ Mass Spectrometry**
**EPA 625**

Facility Name:_____VELAP ID_____

Assessor Name:_____Analyst Name:_____Inspection Date_____

| Relevant Aspect of Standards | Method Reference | Y | N | N/A | Comments |
|---|---|---|---|---|---|
| *Records Examined:* SOP Number/ Revision/ Date _____ Analyst:_____<br>Sample ID: _____ Date of Sample Preparation:_____ Date of Analysis:_____ | | | | | |
| Was glassware heated in a muffle furnace to 400°C for 15-30 minutes after washing **OR** rinsed with acetone or hexane? | 3.1 | | | | |
| Were samples collected in glass containers? | 5.1, 9.1 | | | | |
| Was sodium sulfate purified by heating at 400°C for four hours? | 6.6 | | | | |
| For internal standard calibration, were at least three concentration levels of standards used, with one standard near but above the MDL? | 7.2.1 | | | | |
| Was the working calibration curve or RF verified each working day by the measurement of one or more calibration standards to within ±20%? | 7.3 | | | | |
| Were 5% of samples spiked? (At least one per month.) | 8.1.4, 8.3 | | | | |
| For DOCs, did average recoveries and standard deviations meet the criteria of Table 6 (see attached)? | 8.2.5 | | | | |
| Were samples iced or refrigerated at 4°C from the time of collection until extraction? | 9.2 | | | | |
| Were samples checked for residual chlorine and dechlorinated with 80 mg sodium thiosulfate per liter? | 9.2 | | | | |
| Were samples extracted within 7 days of sampling, and were extracts analyzed within 40 days of extraction? | 9.3 | | | | |
| Was the sample meniscus marked on the bottle for volume determination? | 10.2 | | | | |
| Was each sample adjusted to pH > 11 with NaOH? | 10.2 | | | | |
| Were empty bottles rinsed with 60 mL of methylene chloride, which was then transferred to the separatory funnels? | 10.3 | | | | |
| Notes/Comments: | | | | | |

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| **Separatory Funnel Extraction** | | | | | |
| Were the separatory funnels shaken with venting for two minutes and then the organic layer allowed to separate for at least ten minutes? | 10.3 | | | | |
| If emulsions of greater than one-third the volume of the solvent layer formed, were mechanical techniques employed to complete the phase separation? | 10.3 | | | | |
| Was the methylene chloride layer collected and extracted? | 10.3 | | | | |
| If the emulsion could not be broken, was the sample, emulsion, and solvent transferred to a continuous extractor and that method employed? | 10.3 | | | | |
| Were two more cycles of the extraction procedure repeated on the sample containers and separatory funnels, using two more 60 mL portions of methylene chloride and combining the final extracts? (This is the base/neutral fraction.) | 10.4 | | | | |
| Was the pH of the aqueous phase adjusted to pH < 2 with sulfuric acid, extracted three times with 60 mL methylene chloride, and the final extracts combined? (This is the acid fraction.) | 10.5 | | | | |
| Were both the base/neutral fraction and the acid fraction filtered through a solvent-rinsed drying column containing 10 cm anhydrous sodium sulfate and collected in a Kuderna-Danish (K-D) concentrator? | 10.7 | | | | |
| Were the flasks and columns rinsed with 20-30 mL of methylene chloride to complete the transfer? | 10.7 | | | | |
| Were one or two clean boiling chips added to each K-D fraction, and a three-ball Snyder column attached to each evaporation flask? | 10.8 | | | | |
| Were fractions evaporated in a water-bath at temperatures needed to complete concentration to 1 mL in 15-20 minutes (approximately 60-65°C, adjustable)? | 10.8 | | | | |
| Were the flasks allowed to cool and rinsed into concentrator tube with 1-2 mL of methylene chloride? | 10.8 | | | | |
| Notes/Comments: | | | | | |

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| Were tubes concentrated again with two-ball Snyder columns to 0.5 mL within 5-10 minutes (same temp.)? | 10.9 | | | | |
| Were flasks cooled and rinsed into tubes with about 0.2 mL acetone or methylene chloride, adjusting the volumes up to 1.0 mL with solvent? | 10.9 | | | | |
| If extracts were to be stored longer than two days, were they transferred to Teflon-sealed screw-cap vials? | 10.9 | | | | |
| **Continuous Extraction (For serious emulsion problems)** | | | | | |
| After adding sample to the continuous extractor, was 60 mL of methylene chloride used to rinse sample bottle and added to the extractor? | 11.2 | | | | |
| Was the bottle rinse repeated with 50-100 mL of methylene chloride, and rinsing added to the extractor? | 11.3 | | | | |
| For the base/ neutral fraction, was 200-500 mL methylene chloride added to the distillation flask with enough reagent water to allow for proper operation? | 11.4 | | | | |
| Was aqueous phase adjusted to pH<2 using sulfuric acid and extracted as in 10.6 through 10.9, with 500 mL of methylene chloride added to a clean distillation flask which was then attached to the extractor? | 11.5 | | | | |
| Were extractions performed for 24 hours? | 11.4 | | | | |
| Were dryings, concentrations, and sealings of both the base/neutral fraction and the acid fraction extracts done according to steps 10.6 through 10.9 (see checklist page 2)? | 11.4 | | | | |
| Each day base/neutral fractions were analyzed for benzidine, was the benzidine tailing factor calculated to be less than 3.0? | 12.4 | | | | |
| Each day acids were analyzed, was the tailing factor for pentachlorophenol calculated to be less than 5? | 12.5 | | | | |
| Each day of analysis, was 2 µL of decafluorotriphenyl phosphine (DFTPP) standard solution (25 µg/mL) injected, and were the m/z criteria in Table 9 confirmed before any samples were analyzed? | 12.3 | | | | |
| Were internal standard solutions added to the sample extracts immediately prior to injection? | 13.3 | | | | |
| Notes/Comments: | | | | | |

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| Were 2-5 µL of samples and standards injected into the GC using the solvent-flush technique? | 13.4 | | | | |
| Were extracted ion current profiles (EICPs) obtained? (These are plots of m/z abundances versus time or scan number.) | 14.1 | | | | |
| Did the characteristic masses of each parameter of interest maximize in the same or within one scan of each other? | 14.1.1 | | | | |
| Did the retention time fall within ±30 seconds of the retention time of the authentic compound? | 14.1.2 | | | | |
| Did the EICPs have relative peaks heights of the three characteristic masses which fell within ±20% of the relative intensities of these masses in a reference mass spectrum? | 14.1.3 | | | | |
| Were structural isomers identified as isomeric pairs except when the baseline valley heights between the isomers was less than 25% of the sum of the two peak heights? | 14.2 | | | | |
| Notes/Comments: | | | | | |

Table 9—DFTPP Key Masses and Abundance Criteria

| Mass | m/z Abundance criteria |
|---|---|
| 51 | 30-60 percent of Mass 198. |
| 68 | Less than 2 percent of Mass 69. |
| 70 | Less than 2 percent of Mass 69. |
| 127 | 40-60 percent of Mass 198. |
| 197 | Less than 1 percent of Mass 198. |
| 198 | Base peak, 100 percent relative abundance. |
| 199 | 5-9 percent of Mass 198. |
| 275 | 10-30 percent of Mass 198. |
| 365 | Greater than 1 percent of Mass 198. |
| 441 | Present but less than Mass 443. |
| 442 | Greater than 40 percent of Mass 198. |
| 443 | 17-23 percent of Mass 442. |

Table 6—QC Acceptance Criteria—Method 625

| Parameter | Test conclusion (µg/L) | Limits for s (µg/L) | Range for $\bar{X}$ (µg/L) | Range for P, $P_s$ (Percent) |
|---|---|---|---|---|
| Chrysene | 100 | 48.3 | 44.1-139.9 | 17-168 |
| 4,4'-DDD | 100 | 31.0 | D-134.5 | D-145 |
| 4,4'-DDE | 100 | 32.0 | 19.2-119.7 | 4-136 |
| 4,4'-DDT | 100 | 61.6 | D-170.6 | D-203 |
| Dibenzo(a,h)anthracene | 100 | 70.0 | D-199.7 | D-227 |
| Di-n-butyl phthalate | 100 | 16.7 | 8.4-111.0 | 1-118 |
| 1,2-Dichlorobenzene | 100 | 30.9 | 48.6-112.0 | 32-129 |
| 1,3-Dichlorobenzene | 100 | 41.7 | 16.7-153.9 | D-172 |
| 1,4,-Dichlorobenzene | 100 | 32.1 | 37.3-105.7 | 20-124 |
| 3,3'-Dhlorobenzidine | 100 | 71.4 | 8.2-212.5 | D-262 |
| Dieldrin | 100 | 30.7 | 44.3-119.3 | 29-136 |
| Diethyl phthalate | 100 | 26.5 | D-100.0 | D-114 |
| Dimethyl phthalate | 100 | 23.2 | D-100.0 | D-112 |
| 2,4-Dinitrotoluene | 100 | 21.8 | 47.5-126.9 | 39-139 |
| 2,6-Dinitrotoluene | 100 | 29.6 | 68.1-136.7 | 50-158 |
| Di-n-octyl phthalate | 100 | 31.4 | 18.6-131.8 | 4-146 |
| Endosulfan sulfate | 100 | 16.7 | D-103.5 | D-107 |
| Endrin aldehyde | 100 | 32.5 | D-188.8 | D-209 |
| Fluoranthene | 100 | 32.8 | 42.9-121.3 | 26-137 |
| Fluorene | 100 | 20.7 | 71.6-108.4 | 59-121 |
| Heptachlor | 100 | 37.2 | D-172.2 | D-192 |
| Heptachlor epoxide | 100 | 54.7 | 70.9-109.4 | 26-155 |
| Hexachlorobenzene | 100 | 24.9 | 7.8-141.5 | D-152 |
| Hexachlorobutadiene | 100 | 26.3 | 37.8-102.2 | 24-116 |
| Hexachloroethane | 100 | 24.5 | 55.2-100.0 | 40-113 |
| Indeno(1,2,3-cd)pyrene | 100 | 44.6 | D-150.9 | D-171 |
| Isophorone | 100 | 63.3 | 46.6-180.2 | 21-196 |
| Naphthalene | 100 | 30.1 | 35.6-119.6 | 21-133 |
| Nitrobenzene | 100 | 39.3 | 54.3-157.6 | 35-180 |
| N-Nitrosodi-n-propylamine | 100 | 55.4 | 13.6-197.9 | D-230 |
| PCB-1260 | 100 | 54.2 | 19.3-121.0 | D-164 |
| Phenanthrene | 100 | 20.6 | 65.2-108.7 | 54-120 |
| Pyrene | 100 | 25.2 | 69.6-100.0 | 52-115 |
| 1,2,4-Trichlorobenzene | 100 | 28.1 | 57.3-129.2 | 44-142 |
| 4-Chloro-3-methylphenol | 100 | 37.2 | 40.8-127.9 | 22-147 |
| 2-Chlorophenol | 100 | 28.7 | 36.2-120.4 | 23-134 |
| 2,4-Dichlorophenol | 100 | 26.4 | 52.5-121.7 | 39-135 |
| 2,4-Dimethylphenol | 100 | 26.1 | 41.8-109.0 | 32-119 |
| 2,4-Dinitrophenol | 100 | 49.8 | D-172.9 | D-191 |
| 2-Methyl-4,6-dinitrophenol | 100 | 93.2 | 53.0-100.0 | D-181 |
| 2-Nitrophenol | 100 | 35.2 | 45.0-166.7 | 29-182 |
| 4-Nitrophenol | 100 | 47.2 | 13.0-106.5 | D-132 |
| Pentachlorophenol | 100 | 48.9 | 38.1-151.8 | 14-176 |
| Phenol | 100 | 22.6 | 16.6-100.0 | 5-112 |
| 2,4,6-Trichlorophenol | 100 | 31.7 | 52.4-129.2 | 37-144 |

s = Standard deviation for four recovery measurements, in µg/L (Section 8.2.4).
$\bar{X}$ = Average recovery for four recovery measurements, in µ/L (Section 8.2.4).
P, $P_s$ = Percent recovery measured (Section 8.3.2, Section 8.4.2).
D = Detected; result must be greater than zero.
*NOTE:* These criteria are based directly upon the method performance data in Table 7. Where necessary, the limits for recovery have been broadened to assure applicability of the limts to concentrations below those used to develop Table 7.
[a]The proper chemical name is 2,2'oxybis(1-chloropropane).